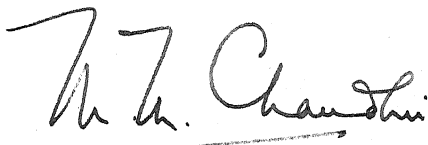
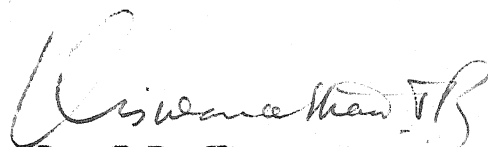


CERTIFICATE

Certified that this work has been carried out under our supervision and that this has not been submitted elsewhere for a degree.



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## LIST OF SYMBOLS

Q	-	Rate of gas removal
S	-	Pumping speed
P	-	Equilibrium pressure in the system
n	-	Overtone number
C	-	Shear elastic constant of the quartz crystal
t	-	Thickness of the crystal
$\rho$	-	Density of quartz
N	-	Constant, (for AT-cut crystal $N = 1670$ kc/s mm)
$f_n$	-	Unperturbed frequency of resonance of quartz crystal
$f_{pn}$	-	Perturbed frequency of resonance of quartz crystal
$\rho_d$	-	Density of the deposit
A	-	Area of the film deposited on the crystal (monitor)
m	-	Total mass deposited on the monitor crystal
K	-	Mass distribution constant (approximately unity)
$\Delta f$	-	Frequency shift
$C_f$	-	'Mass determination sensitivity' constant of the crystal

## Abbreviations

$\text{\AA}$	-	Angstrom
c/s	-	cycles/second
UHV	-	Ultra-high vacuum
KVA	-	Kilo volt-ampere

## SYNOPSIS

### THIN FILM DEPOSITION AND THICKNESS MEASUREMENT

F.C. Jain

An ultra-high vacuum evaporation unit is set up for the deposition of thin films of controlled thickness. Pressures measured are of the order of  $10^{-7}$  -  $10^{-8}$  torr inside the evaporation chamber. A quartz crystal monitor (with water-cooled crystal holder) is designed to measure the rate of deposition and the thickness of the thin film. The frequency of resonance of the monitor crystal shifts when it is mass loaded by the evaporant. This shift being linearly proportional to the mass loading, provides information about the thickness and the rate of deposition of the thin film. The mass determination sensitivity of the monitor is  $2.78 \times 10^7$  for copper deposits. It corresponds to a frequency shift of 10 c/s for  $3.6 \times 10^{-7}$  gm/sq cm.

INTRODUCTION

## 1.1        General Considerations

Thin films have been the subject of extensive investigations ever since the first films were fabricated more than hundred years ago. However, the rapid growth of thin film technology started between the two World Wars when vacuum evaporated metallic and dielectric films were used for mirrors and antireflection coatings. The development of diffusion pump made possible the construction of large vacuum systems, and vacuum evaporation apparatus began to be used in the optical industry. Since then, thin films of metals, semiconductors, and dielectrics have become increasingly important for fundamental studies in many fields such as physics, electronics, and chemistry and are also employed in numerous practical applications. The tremendous progress in the field of thin film physics has been stimulated by the recent development of highly efficient ultra-high vacuum systems in which thin films can be deposited by vacuum evaporation techniques. The advanced instrumentation for measuring pressure, deposited mass during evaporation, and automatically controlled evaporation sources, now permit the fabrication of thin films under controlled and reproducible conditions.

There are many practical considerations which make it profitable and sometimes even necessary to use thin films. For

example, thin films provide the only form in which alkali metals in a state of high purity can be used for studies of photoelectric phenomena and electrical conductivity. The use of thin films for the preparation and study of surfaces appears attractive. Microcircuits, computer memories, insulated-gate field effect transistors, superconducting devices, and various other devices when made of thin film are not only compact but also exhibit an improved performance. A superconducting thin film of  $\text{Nb}_3\text{Sn}$  deposited on a niobium base provides very high critical current densities.

## 1.2 Basic Properties of Thin Films

Investigations on the basic properties of the thin films can be grouped into two categories. The first is concerned with the physical properties which primarily depend upon the thickness of the film. This is due to the fact that electrons mean free path and relaxation time are functions of film thickness. By "thin" we mean the thicknesses in the range of few angstroms to 0.1 micron. The effect of mean free path of electrons in the electrical conduction of thin metal films, studies of the magnetization of thin films of ferromagnetic materials as a function of film thickness, tunneling effects, and the effects due to the adsorption of gasses are examples of this class.

In some cases the properties are least dependent upon the thickness of the film, but are mainly dictated by the structure of the film. The study of the thin film structures

makes up the second category. The structure of the film depends upon its nucleation and growth conditions. The film structure is frequently quite different from the bulk material.

### 1.3 Role of Vacuum in Thin Film Fabrication

Vacuum in which film is deposited plays an important role in the determination of the physical properties of the thin film. The properties of thin films, in some cases, are very sensitive to the presence of certain residual gases in the system. For instance, it is difficult to prepare thin, tantalum films with superconducting properties similar to those of bulk material because these properties are extremely sensitive to the presence of common residual gases such as nitrogen, hydrogen, and oxygen<sup>1</sup>. The contamination of the film due to a specific gas during its deposition is proportional to partial pressure of that gas in the chamber. Thus the films deposited in better vacuum are less susceptible to contaminations. It can be shown that in vacuum of the order of  $10^{-6}$  torr, it takes 1.3 secs. to form a monolayer of gas on a clean surface. However, if the film is deposited in a vacuum of the order of  $10^{-8}$  /  $10^{-9}$  torr, it takes approximately 20 minutes to form a monolayer of the gas on a clean surface, time during which meaningful observations could be carried out. Film evaporation and deposition, therefore, has to be carried out in vacuum of the order of  $10^{-8}$  /  $10^{-9}$  torr or better.

#### 1.4 Vacuum Deposition of Thin Films

For the above mentioned reasons thin films are always deposited in high or ultra-high vacuum conditions and their thickness measurements are done during deposition. Various techniques are in use for the deposition of thin films in vacuum conditions. The choice of a deposition technique depends upon the material to be deposited and the ultimate use. For instance, vacuum evaporation and cathode sputtering are the widely used processes for fabricating thin film passive and active elements in microelectronic assemblies. Vacuum evaporation technique is the one used in the present work for depositing thin films. A vast number of metals and non-metals can be evaporated to give thin film of controlled thickness, composition and structure.

#### 1.5 Aim of the Work

The aim of the present work is to simulate conditions for the deposition of thin film of controlled thickness in high/ultra-high vacuum conditions. The work centres around three main areas:

- (I) Setting up of a pumping station to attain pressures of the order of  $10^{-8}$  torr. This is done by making use of existing Granville-Phillips pumping unit with adequate alterations.
- (II) Fabrication and building up of an evaporator assembly which consists of designing and construction of the base plate and the evaporation chamber.

(III)

Fabrication of the thin film thickness measuring unit. Quartz crystal monitor is designed and fabricated to measure the film thickness. There is a shift in the frequency of resonance of the monitor crystal when it is mass loaded by deposition. The mass and time deposition give the information about film thickness and rate of evaporation.

Copper has been used as an evaporant in film thickness measurements.



## CHAPTER TWO

### THE ULTRA-HIGH VACUUM SYSTEM

#### 2.1 Introduction

The pressure in a vacuum system depends upon the rate of removal of the gas liberated in the system from external as well as internal sources. In a properly constructed system, real leaks from the atmosphere into the system are negligibly small. Thus, the gas load which the pumps must handle at low pressure comes from gases or vapours desorbing from the surface of parts inside the system or from the pumps. The ultimate pressure in a vacuum system is attained when the rate of gas removal equals the rate of inflow from all the sources<sup>2</sup>. The rate of gas removal expressed in terms of pumping speed and the equilibrium pressure is given by

$$Q = S \cdot P \quad \dots \quad (2.1)$$

where  $Q$  is the rate of gas removal,  $S$  the pumping speed and  $P$  the pressure in the system. So the order of vacuum can be increased by increasing the speed of pumping and minimizing the gas inflow into the system. The gas liberated or desorbed from within the system depends upon the material used<sup>3</sup>. The material, therefore, should satisfy the following requirements: it should have negligible vapour pressure at the highest operating temperature; adsorption of the gases from the surface of the material should be as small as possible; permeation of the gas through the material should be negligible.

## 2.2 Vacuum Pumping Station

Granville-Phillips pumping equipment is used to achieve ultra-high vacuum for the evaporation chamber. The schematic of the pumping station is shown in Fig. ( 1 ).

### 2.21 Fore Pump:

A Duo-Seal rotary oil pump with 100 litres/min. pumping speed is used to reduce the pressure in the system up to 1 micron.

### 2.22 Diffusion Pump:

The fore pump alone will not bring down pressures below 1 micron ( $10^{-3}$  torr), hence, a water cooled oil diffusion pump is also put in series. The one used in the system is a three stage fractionating diffusion pump.

### 2.23 Liquid Nitrogen Cold Trap (Cryosorb):

The cold trap is provided to prevent oil vapours from diffusing into the system without seriously impeding the flow of molecules from the system into the pump. In addition, it aids in pumping significantly by condensing gases on to its surface.

## 2.3 Controls

### 2.31 Front Panel Control:

Two switches are provided on the front panel for the operation of the fore pump and the diffusion pump respectively. An electrical interlock prevents 'turning on' of the diffusion pump if the fore pump is not switched on. A water flow switch and an air flow switch are connected in series with the diffusion

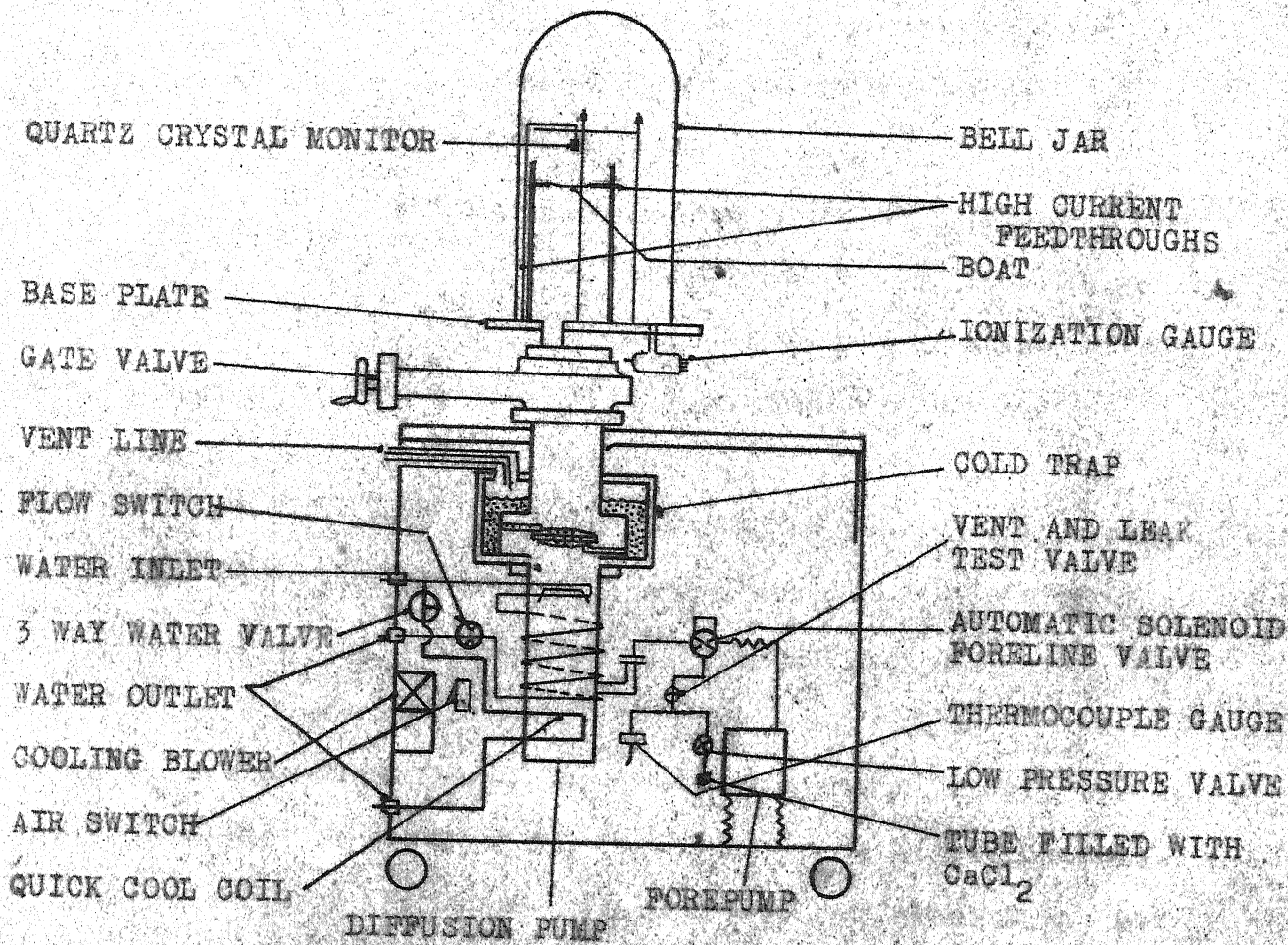


FIG. 1  
SCHEMATIC OF THE PUMPING STATION AND THE EVAPORATION UNIT

pump switch. Thus, the diffusion pump cannot be turned on if the supply of either water or air is inadequate.

#### 2.32 Foreline Solenoid Valve:

An automatic electrically operated solenoid valve is incorporated in between the diffusion pump and the fore pump. This valve seals off the diffusion pump whenever the fore pump is shut off or when power failure occurs.

#### 2.33 Cooling Water Valve:

Provision for quick cooling of the diffusion pump is made by providing a water valve which in its vertical position routes the water through quick cool coil.

#### 2.34 Thermocouple Gauge Port and Vent Valve:

An arrangement has been made, shown in Fig. ( 1 ), to measure pressure in the foreline and to allow controlled dry air venting into the system. This arrangement was not incorporated in the unit. A T-joint is connected to the vent valve. Thermocouple gauge is mounted on one end of this tube and on the other end a low pressure valve is silver soldered. The other side of the low pressure valve is connected to an open tube filled with calcium chloride.

#### 2.4 The CVC Gate Valve

The gate valve is mounted on the flanged manifold port of the vacuum system, as shown in Fig. ( 1 ). A base plate, described in Section 2.5, is mounted on the top of the gate valve. Viton 'O' rings are sandwiched for vacuum sealing between flanged manifold port, gate valve, and base plate. The gate valve

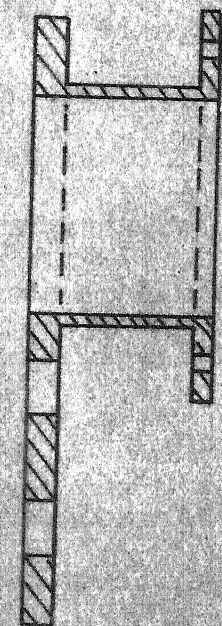
isolates the evaporation chamber mounted on the base plate from the vacuum system.

## 2.5 Base Plate

The base plate is an important part of the ultra-high vacuum evaporator unit. It provides coupling between evaporation chamber and the vacuum system.

The base plate is made up of mild steel and is first coated with copper and then dull nickel. The mild steel as the material of base plate was chosen because stainless steel plate of desired thickness ( $3/4"$ ) was not available. The base plate consists of two circular plates A and B. These are welded together with a cylinder placed in between them eccentrically. This is shown in the Fig. ( 2 ). The smaller plate B has eight holes matching with tapped holes in the gate valve. On the other hand the upper plate has nine holes for various feedthroughs. Six blind tapped holes are provided for each feedthrough on the back of the plate A to screw in the feedthroughs inside the chamber. Plate A also has three tapped blind holes on top of it for tripod stand in the evaporation chamber.

The evaporation chamber is described in detail in Chapter 3.



UPPER BASE PLATE (A)

CYLINDER

LOWER BASE PLATE (B)

FIG. 2

SECTIONAL VIEW OF THE BASE PLATE

## CHAPTER THREE

### THE EVAPORATION UNIT

#### 3.1 Introduction

High vacuum evaporation is the simplest and precise technique of producing thin film of material on to a suitable substrate such as glass, mica, or sodium chloride crystal etc. It works well with material of different types (metals and non-metals) and can be used to give surface film of large area.

A high vacuum evaporation unit to nucleate thin film of controlled thickness is described below. The schematic of the evaporation unit is shown in Fig. ( 3 ).

#### 3.2 Description

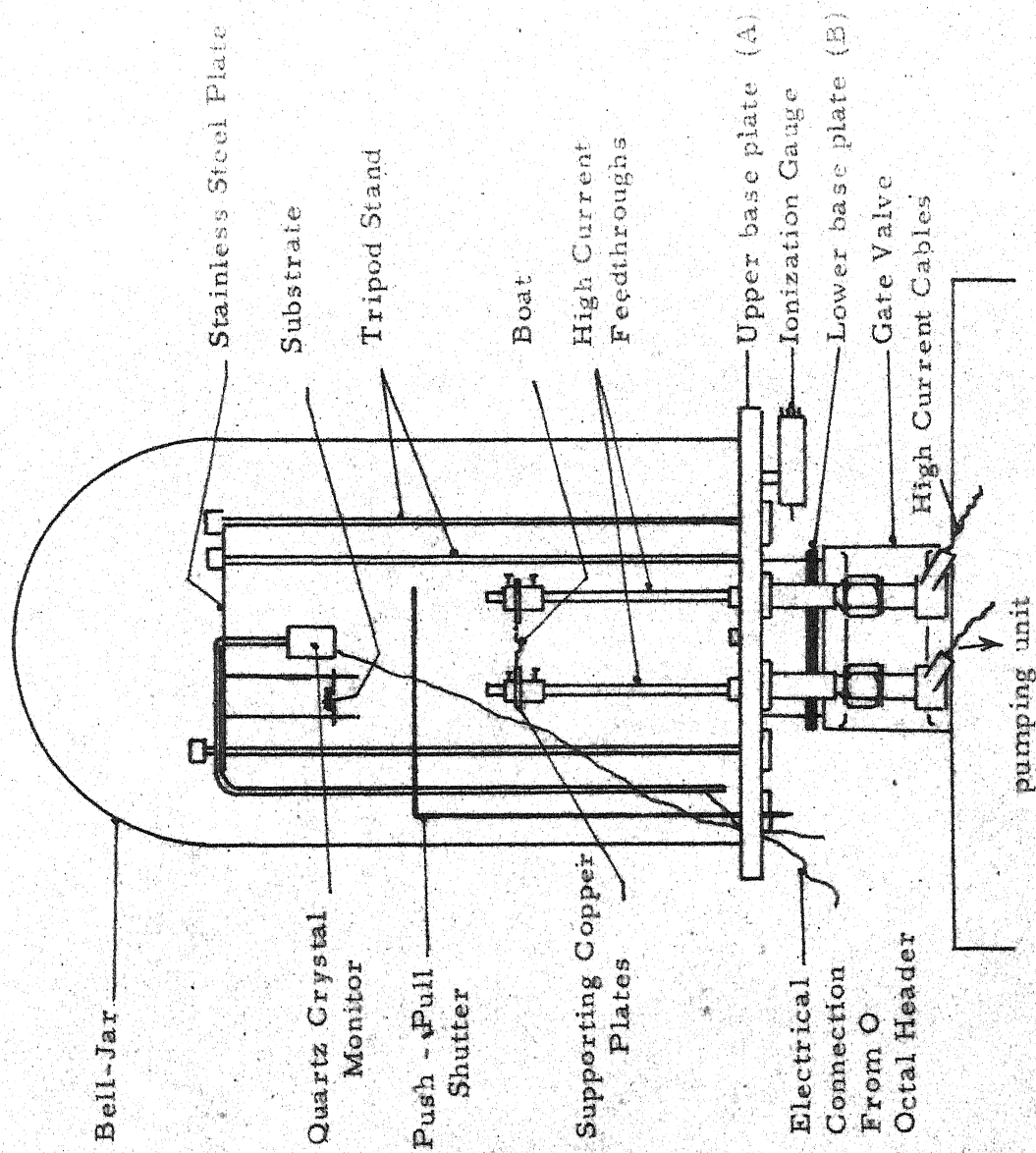
The various components of the evaporation unit are discussed according to their functions. The base plate, on which the whole evaporation assembly is mounted, has already been described in 2.5.

##### 3.21 Pressure Measuring Device:

Pressures below 1 micron are measured by means of a thermocouple gauge mounted in the foreline of the vacuum system, and is already discussed earlier. Pressures in  $10^{-4}$  -  $10^{-10}$  torr range are measured by Veeco ionization gauge. An arrangement is made, as shown in Fig. ( 4 ), to mount the ionization gauge on the base plate. Two viton 'O'-rings are used in the arrangement for sealing.



Fig. 3 Schematic of  
EVAPORATION UNIT





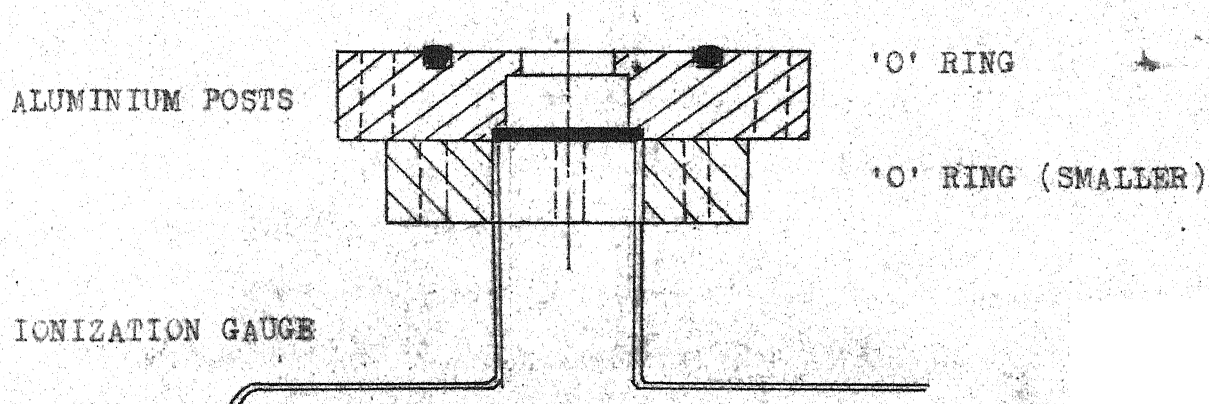


FIG. 4

IONIZATION GAUGE MOUNT

### 3.22 Evaporation Source and its Accessories

The material to be evaporated is supported by boat made up of refractory metal (W, Mo, Ta, Nb) foil which also serves the purpose of heating element. The material of the boat is chosen so that it does not react with the evaporant and cause contamination of the film deposited. For instance, in case of copper, the prescribed boat materials are tantalum (Ta) and molybdenum (Mo)<sup>4</sup>. Molybdenum boat has been used for the evaporation of copper in the system. The boat is tightly supported by thick copper plates which are rigidly clamped to the high current feedthroughs. The temperature of the evaporant is raised by the electric resistance heating of the boat. The electric circuit diagram of the heating arrangement is shown in Fig. ( 5 ).

Power to the input of 4 KVA variable transformer  $T_1$  is supplied from the a.c. mains. The controlled output power from transformer  $T_1$  is fed through an ammeter A to the primary of 2 KVA stepdown transformer  $T_2$  designed for 220/230 volts operation. Transformer  $T_2$  provides high currents at low voltages. Secondary of transformer  $T_2$  could be tapped for 5V, 10V, 20V and 40 volts depending upon the load, and supplies power to the boat (or filament). The temperature of the boat is controlled by adjusting the setting of the transformer  $T_1$ . Ammeter A and the transformer  $T_1$  are calibrated in terms of the temperature of the boat with the help of an optical pyrometer (Leeds-Northrup type) which gives direct temperature measurements.

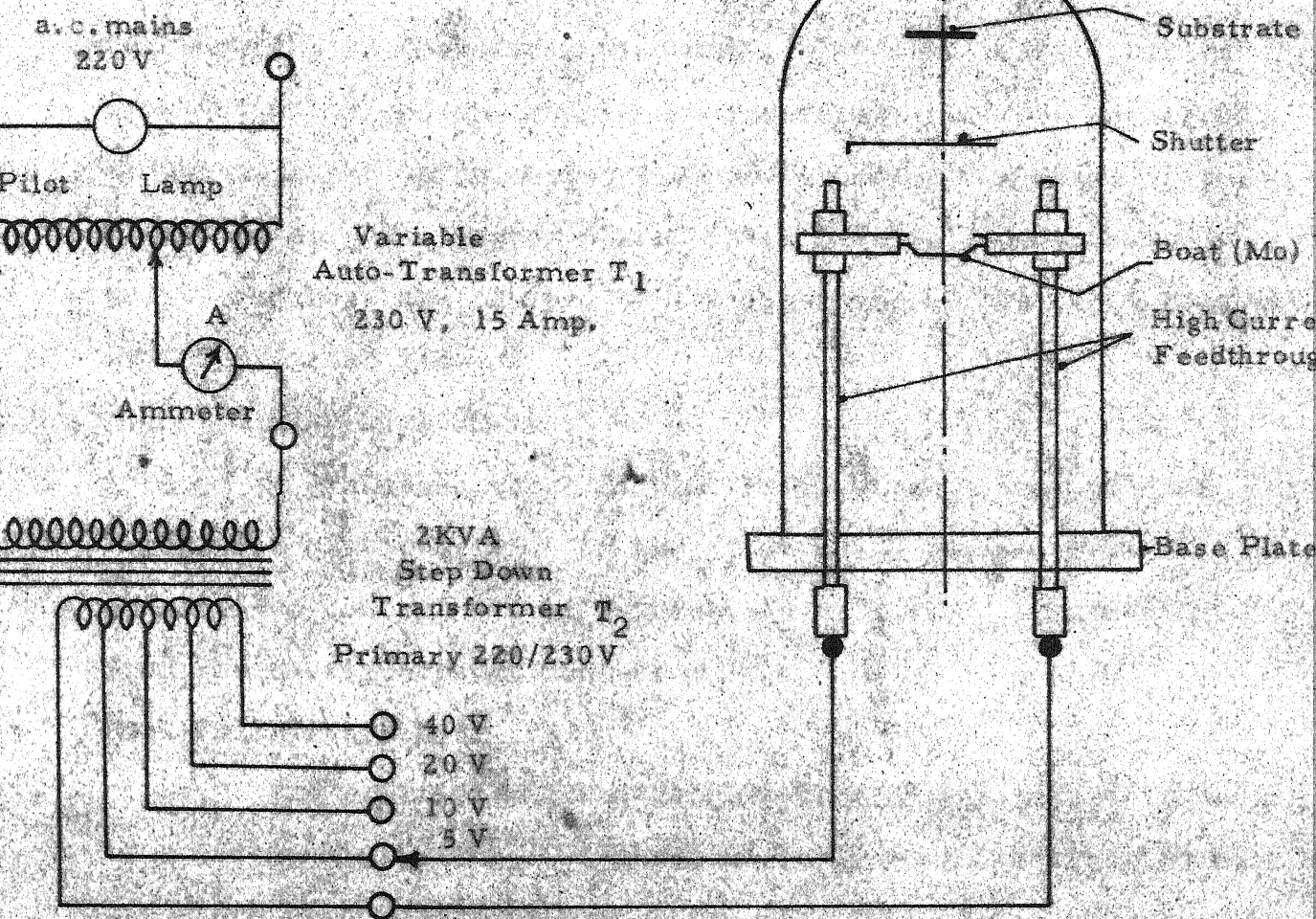


Fig. 5 Schematic Circuit Diagram of the Heating Arrangement.

## High Current Feedthroughs:

The arrangements are made to use two MRC high current feedthroughs in the evaporation unit. Two high current cables from the output of the step down transformer  $T_2$  are connected to two T-shaped thick copper sleeves fitted on the stems of the feedthroughs. The copper plates which provide support for molybdenum boat are clamped to the upper end of the feedthroughs by thick copper sleeves.

### 3.23 Push-Pull Shutter:

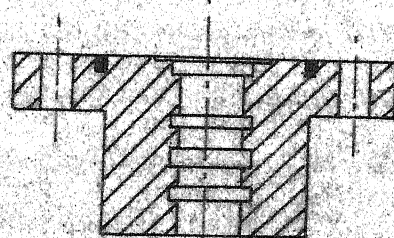
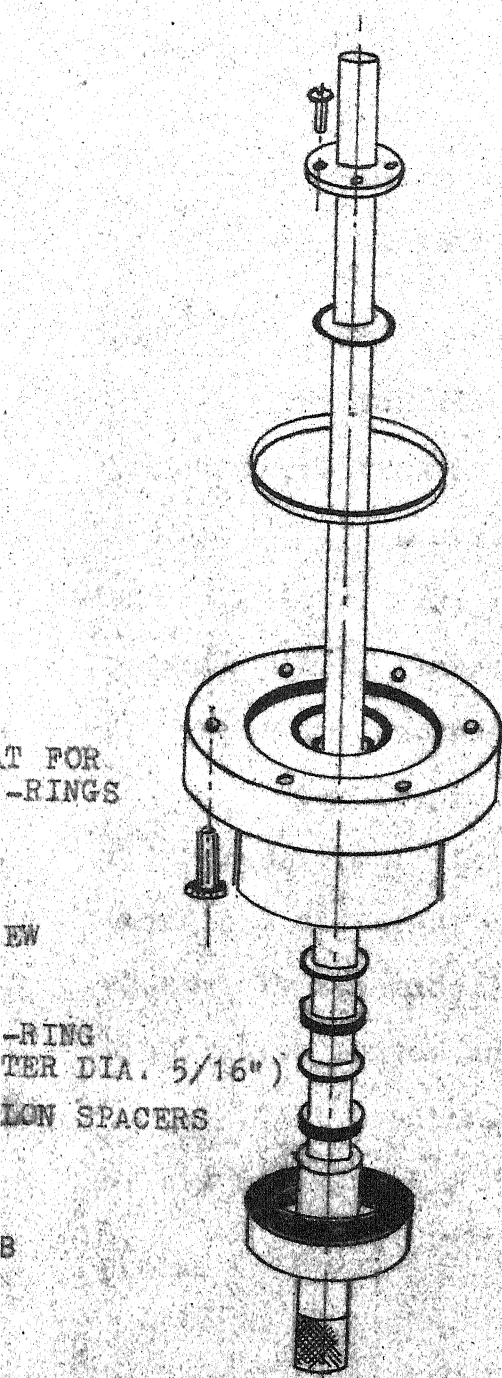
A stainless steel shutter is provided to control the deposition of the film on to the substrate. It can also be operated to control the film thickness by getting information from the quartz crystal monitor unit.

### Push-Pull Rotary Rod:

The exploded and the section view of the push-pull rod is shown in Fig. ( 6 ). It has been designed and fabricated to withstand high vacuum without leakages. Three Viton 'O' rings are used at different sections of the stainless steel rod. These are kept in place by teflon spacers and used to vacuum seal the feedthroughs. Continuous rotary motion, in either direction, and 8" of vertical linear motion can be transmitted to the interior of the bell-jar by means of the push-pull rotary.

### 3.3 Thin Film Thickness Measuring Device

The thickness of the thin film is measured in situ while the film is being deposited. The thickness is measured by means of quartz crystal monitor discussed in detail in



SECTION VIEW OF  
SEAT FOR 'O'-RINGS

FIG. 6

EXPLODED AND SECTION VIEW OF THE PUSH-PULL ROD

Chapter 4. The two leads from the monitor crystal are soldered to two contacts of the octal header. The crystal holder is supported inside the chamber by the tripod stand.

#### MRC Octal Header:

It provides eight low voltage connections to devices inside the bell-jar.

### 3.4 Substrate Mount

The importance of the substrate cannot be over-emphasized in the deposition of thin films. The major properties to be considered are surface characteristics, chemical composition and stability.<sup>5</sup> The range of materials presently available includes organic films, glasses, ceramics, single crystal substances. The important surface properties are flatness and microfinish.

Microscopic glass slides are used as substrate for thin film deposition. This is mounted on a stainless steel plate which has different holes, and which is supported inside the chamber by two rods. The distance between substrate and evaporation source can be adjusted.

(All feedthroughs are mounted below the base plate A by six screws and a Viton 'O' ring.)

The monitoring device must be reliable and stable in its response over long periods of deposition, and should not be susceptible to variations of temperatures, pressures, and mechanical shocks during deposition. Monitored information about thickness or rate of deposition must not depend upon the



physical properties of the film, which may change randomly. Monitoring sensitivity should be independent of the deposition rate and the film thickness. Measurements on both dielectric and metallic films should be possible. The monitoring device should be adaptable to a feedback control system to allow automatic control of evaporation rate.

#### 4.22 Types of Thin Film Monitors:

Thin film monitors, which give information about rate of evaporation/deposition and thickness of the film during the deposition process, are fundamentally of three types: (i) monitors which depend on the mass loading of the monitor element; (ii) monitors which depend on the flow of evaporant through an electrode arrangement; (iii) and lastly the monitors which are sensitive to the impact of deposit on a sensitive surface.

Instruments based on mass loading principle give the mass of the film deposited, while the instruments of second and third type give information about the rate of deposition of the film. Since the rate of evaporation and thickness are interrelated, hence, in principle, it does not matter which quantity is measured; the rate can be obtained from mass or thickness measurements by differentiation; while thickness (or mass) of the film deposited can be obtained from rate measurements by integration.

Quartz crystal monitors, ionization monitors, and microbalances are the illustrated examples of these three classes respectively. We shall be concerned with quartz crystal monitors only.<sup>6</sup>



#### 4.3 Quartz Crystal Monitors

Robust construction together with very high "mass determination sensitivity" makes quartz crystal monitors, the obvious choice over other types of monitors. Measuring the mass of the thin film deposit using quartz crystal monitors, is based on measuring shift in the frequency of resonance of a quartz crystal oscillator with mass loading when crystal is vibrating in thickness shear mode. Quartz crystal monitors satisfy aforementioned requirements very satisfactorily over a fairly large range of the film thickness.

##### 4.31 Monitor Crystal Selection:

Depending on the types of cut, a quartz crystal can be made to resonate in different modes. The basic idea in selecting a type of cut is that the major surfaces of the crystal shall be antinodal. When the major surfaces of the crystal are antinodal the added mass, either on one or both sides, will shift the frequency of resonance of the crystal. In thickness shear mode the two major faces of the crystal are antinodal. Y, AT and BT-crystal cuts vibrate in thickness shear mode.<sup>7</sup>

Fig. (7) shows popular orientation of the two high frequency cuts and their various characteristics (e.g., variation of resonant frequency with temperature). AT-cut is preferred because of its small temperature coefficient and superior frequency-temperature characteristics. AT-cut commercially available crystals with zero temperature coefficient at room temperature are used in the present work.

Having selected the crystal cut, its frequency of resonance is so chosen that it gives optimum "mass determination sensitivity". This is discussed in the next section.

#### 4.32 Theoretical Relation between Mass Loading and Frequency Shift:

The frequency resonance for AT-cut crystal is given by

$$f = \frac{n}{2t} \left( \frac{C}{\rho} \right)^{\frac{1}{2}} ; n = 1, 3, 5 \quad (1)$$

where  $n$  is overtone number,  $C$  the shear elastic constant,  $\rho$  the density and  $t$  is the thickness of the crystal<sup>8</sup>. As is obvious from (1), frequency of resonance  $f$  depends upon  $t$ , or  $C$ ; and hence any external influence causing variation in any of these parameters will significantly change it. For instance temperature of crystal, temperature gradients, electric excitation field and hydrostatic pressure will influence  $f$  in so far as they effect either  $t$ ,  $\rho$  or  $C$ .

For AT-cut crystals,  $\frac{1}{2} \left( \frac{C}{\rho} \right)^{\frac{1}{2}} = N = 1670 \text{ Kc/s mm}$ ;  
the value of  $N$  for BT-cut crystals is  $2500 \text{ Kc/s mm}$ .

Change in the frequency of resonance of the crystal due to mass loading has been calculated from perturbation analysis.

If  $f_n$  is the unperturbed frequency, and  $f_{pn}$  is the perturbed frequency ( $n^{\text{th}}$  overtone) of the vibrating crystal,  $f_{pn}$  may be expressed by

$$f_{pn}^2 = f_n^2 \times \frac{1}{1 + \frac{2 K n m}{A \rho_d t}} \quad (2)$$

where  $t$  is the thickness of the resonating quartz plate,  $\rho_d$  is the density of the deposit,  $m$  the total mass added over an

Considering fundamental mode ( $n = 1$ ) we have,

$$f_p^2 = f^2 \times \frac{1}{1 + \frac{2 K m}{A \rho_d t}} = f^2 \left( 1 + \frac{2 K m}{A \rho_d t} \right)^{-1} \quad (3)$$

For deposited mass of the order of 100 micro-grams,  $\frac{2 K m}{A \rho_d t} \ll 1$ ,

Under these conditions,

$$f_p \approx f \left( 1 - \frac{K m}{A \rho_d t} \right).$$

Let the frequency shift be  $\Delta f$ . Thus

$$\frac{\Delta f}{f} = \frac{-K m}{A \rho_d t} \quad (4)$$

From (eqn.4), (eqn.1) and the definition of  $N$  we get

$$\frac{\Delta f}{f^2} = - \frac{K m}{A \rho_d N}$$

$$\text{or} \quad \frac{m}{A} = - \frac{\rho_d N}{K f^2} \Delta f = - \frac{\Delta f}{C_f} \quad (5)$$

where  $C_f = \frac{f^2 K}{N \rho_d}$  is a constant for a given crystal and deposit and is termed as the "mass determination sensitivity" of the crystal. Negative sign indicates that mass loading causes a reduction in frequency.

#### 4.33 Criteria for selecting Oscillator Frequency:

From (eqn.5) and (eqn.1) we note that mass determination sensitivity is proportional to the square of frequency, but the thickness of the crystal is inversely proportional to it. Crystal should not be too thin for ease of handling and on the other hand should give adequate mass determination sensitivity. 6.5 Mc/s crystal is chosen for the work as a reasonable compromise.

Mass determination sensitivity of the crystal vibrating at 6.5 Mc/s is calculated to be  $2.78 \times 10^7$  for copper deposits.

For a deposit build up of 1 micro-gram / sq. cm, the frequency shift is 27.8 c/s.

#### 4.34 Crystal Holder Design ;

To make measurements of frequency shift in the frequency of resonance of the vibrating crystal, the crystal should be so mounted as to be least sensitive to possible heating by radiation from an evaporation source.<sup>10</sup> To keep the crystal temperature stabilized, a crystal holder, as shown in Fig. (8) , is designed.

The 6522.9 Kc/s quartz crystal A, 1.5 x 1 sq. cm face area and approximately 1mm thick is mounted in intimate contact with the water cooled main copper block B. Copper block B forms one electrode and the contact to the other electrode is made by soldering a wire to the annular copper ring C. Teflon disc D isolates the two electrodes. Spring H provides the adjustable mechanical pressure. Temperature of the holder is stabilized by circulating water through copper tubing F. A copper plate E is attached on the front face of the main block B with screws and a stainless steel plate G is mounted on it. The stainless steel plate G contains three different size holes arranged so that any one hole can be placed in front of the crystal, exposing only a desired area to the evaporant.

The crystal holder described above has the following advantages:

- (1) crystal is in intimate contact of a continuously cooled surface;

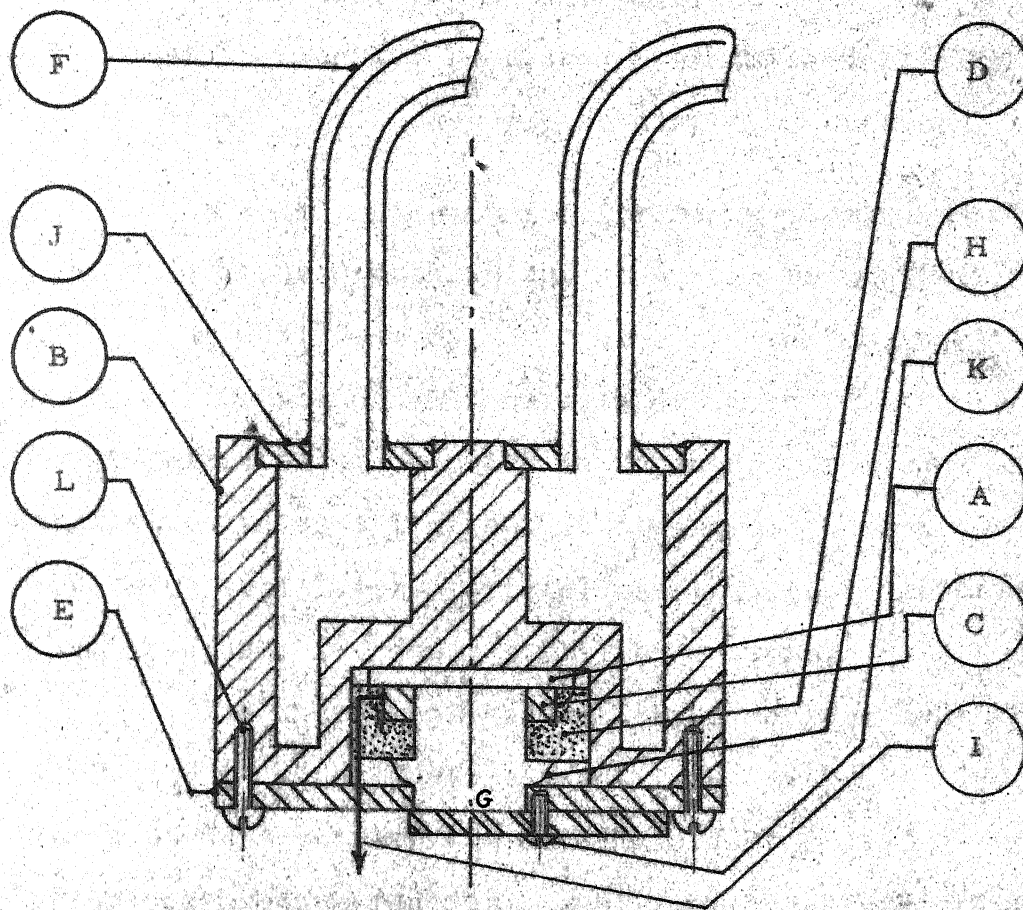


Fig. 8 Sectional View of the Water Cooled Crystal Holder.

S.No	Description	Material
A	Monitor Quartz Crystal.	Quartz
B	Water Cooled Copper Block	Copper
C	Annular Electrode	Copper
D	Insulating Disc	Teflon
E	Heat Shielding Disc	Copper
F	Tubes for Water Cooling	Copper
G	Aperture Adjusting Disc	Stainless Steel
H	Spring (For Adjusting Mechanical Pressure on X <sup>l</sup> tal)	Copper
I	Connection Wire From Copper Ring C	Copper
J	Copper Disc (Silver Soldered to Block B & Tubes F)	Copper
K	Screw	Steel
L	Screw	Steel

- (ii) it can safely be used in ultra high vacuum work;
- (iii) as the crystal is pressure sandwiched it allows the use of plated as well as nonplated crystals.

#### 4.4 Electronic Equipment for Quartz Crystal Monitor

Fig. ( 9 ) shows the schematic block diagram of the monitoring unit developed. Quartz crystal monitor is electrically connected to the monitor oscillator circuit with co-axial cable. The information about shift in resonance frequency of the monitor crystal, with mass loading, is obtained by comparing it with a reference oscillator. Crystal oscillator is preferred as reference oscillator for frequency stability reasons.

The outputs of reference and monitor crystal oscillators are mixed in a nonlinear element. An initial difference of about 25 Kc/s between two oscillators is proved to be useful for mixer performance reasons. The frequency of reference and monitor oscillators are chosen to be 6547.9 Kc/s and 6522.9 Kc/s respectively.

The mixer output is filtered in an active filter with sharp high cut-off frequency characteristics. The cut-off frequency of the filter is chosen so that the filter response is flat up to 200 Kc/s, the maximum signal frequency. Its cut-off frequency is about 315 Kc/s and it gives -36 db/octave fall at the high cut-off end. Thus it effectively rejects the 6.5 Mc/s and other undesirable harmonic contents.

The output of the filter is fed to the discriminator. The discriminator consists of an amplifier, clipper, differentiator, clipper and monostable multivibrator stage. Well shaped and timed

pulses from monostable multivibrator are fed to the counter and to the d.c. averaging meter. When there is no mass loading of the monitor crystal we get a 25 Kc/s pulse train because of the initial difference in the two oscillator frequencies at the output of the monostable multivibrator. And this when averaged in a d.c. meter indicates a voltage reading. So the meter reading is scaled to zero initially. As the monitor crystal is mass loaded, the frequency of one shot pulses increases above the initial value of 25 Kc/s. Since the amplitude and duration of one shot pulses are independent of the triggering signal frequency, there is a linear increase/decrease in meter reading with an increase in pulse frequency. The meter reading decreases if the incoming pulses are of negative polarity. The meter can be directly calibrated in terms of film thickness.

The rate at which the meter reading changes gives the rate of evaporation of the deposit. We get an output voltage proportional to the rate of evaporation by differentiator.

#### 4.41 Crystal Oscillator:

The oscillator circuit described below is used for both monitor and reference oscillators. The circuit diagram shown in Fig. (10) is a modified version of Colpitts Oscillator.<sup>11</sup> The crystal is operated in series resonance mode. The frequency stability of the oscillator is of the order of 2 parts in million over supply voltage range from -6 - -30 volts.

The circuit is designed for 1 ma emitter current. The resistors  $R_1$ ,  $R_2$  provide the bias voltage at the base of the transistor  $T_1$ , and the emitter current is determined by the voltage drop across  $R_3$ .

The frequency of oscillation is adjusted by variable inductors  $L_1$  and  $L_3$ , which consequently permit the use of the same circuit for both reference and monitor oscillators.  $C_2$  is the bypass capacitor for emitter resistor  $R_3$  and  $C_4$  is the decoupling capacitor for the d.c. supply. The capacitor  $C_1$  together with capacitor  $C_3$  provides the necessary feedback in the circuit. The transistor type CIL911 was chosen to be used in the circuit and its alpha-cut-off frequency is approximately 15 times the frequency of the oscillation providing good frequency stability for the circuit. The circuit yields an output voltage of 1V across a load resistance of 2.2K. Temperature variation from  $-5^{\circ}\text{C}$  to  $55^{\circ}\text{C}$  produces a frequency shift of 5 parts in million.

The signals from the monitor and reference oscillators are compared in the mixer described in the next section.

#### 4.42 Mixer:

Fig. (11a) shows the block diagram and Fig. (11b) shows the circuit diagram of the mixer unit. Signal currents from the monitor and reference oscillators are linearly added in the input circuit of the common base stage  $T_1$ . The output from the collector of  $T_1$  being a high impedance source is transformed to a low impedance output using an emitter follower  $T_2$ .

The mixing is done using the nonlinear  $v-i$  characteristic of the emitter-base junction of the transistor  $T_3$  operated in the common emitter mode. The emitter and collector currents of transistor  $T_3$  contain sum and difference frequencies and other



harmonics. The difference frequency which contains the useful information is recovered by feeding the output from the collector of transistor  $T_3$  to a low pass filter. The difference frequency ( $f_{\text{reference}} - f_{\text{monitor}}$ ) is about 25 Kc/s for no mass deposited on the monitor crystal. The difference frequency increases as the monitor crystal is mass loaded or the thickness of the deposited thin film increases. An initial difference of 25 Kc/s between two oscillators is kept for the mixer performance reasons.

#### 4.43 Low Pass Filter:

An active filter is preferred to conventional L-C tuned circuits for better frequency selectivity of the former. Fig. (12) shows the fundamental and complete electrical circuit of the low pass active filter.<sup>12</sup>

Theory of operation:

A section of the filter consists of two high cut-off RC sections coupled by unit gain buffer amplifiers  $A_1$  and  $A_2$ , as shown in Fig. (12a). Output signal  $E_{\text{out}}$  is fed back through an amplifier of gain  $A_3$  to the capacitor  $C_1$  of first high cut-off section. The loop gain of the circuit is  $A_3$ .

The transfer function of the circuit, for  $C_1 = C_2 = C$  is

$$\frac{E_{\text{out}}}{E_{\text{in}}} = \frac{1}{j^2 \omega^2 (R_1 C R_2 C) + j \omega [R_2 C + (1 - A_3) R_1 C] + 1} \quad (6)$$

At the cut-off frequency  $\omega = \omega_{co}$ ,

$$j^2 \omega^2 R_1 C R_2 C + 1 = 0 \quad \text{and} \quad \omega_{co} = \frac{1}{\sqrt{R_1 R_2 C}}$$

the gain of the circuit is  $G_{co} = \frac{1}{\sqrt{R_2/R_1 + (1-A_3)\sqrt{R_1/R_2}}}$  (7)

Eqn. ( 7 ) indicates that the gain of the filter at cut-off can be adjusted by controlling  $A_3$ . Plot of  $G_{co}$  versus frequency is sketched in Fig. (13) for various values of  $A_3$ . Curve 2 is the one used in the filter. It can be obtained if we set  $R_1 = R_2$  and  $A_3 = 0.8$ . That is, if the loop gain of the circuit is 0.8, we get desired filter characteristic. This, for a 12 db/octave section, gives  $G_{co}$  of approximately -0.5 db.

#### Practical Circuit:

Two emitter followers are used in place of amplifiers  $A_1$  and  $A_2$  in the practical circuit. The gains of  $A_1$  and  $A_2$  are of the order of 0.9. No amplifier is used for  $A_3$ . The output of the emitter follower  $A_2$  is directly fed back to the capacitor of the first high cut-off RC section of the filter. The overall loop gain of the circuit is always less than unity and thus ensures good stability of the circuit. The loop gain is about 0.8 and is not of much importance to us, if it is slightly less, because the output of the filter is amplified in the discriminator unit.

The cut-off frequency of the high cut-off section is chosen so that the filter response is flat up to 200 Kc/s, the maximum signal frequency.

If  $R_1 = R_2 = 3.3K$  ,  $C_1 = C_2 = 150$  pf , we get

$$F f_{co} = \frac{1}{2 \times 3.3 \times 10^3 \times 150 \times 10^{-12}} = 315 \text{ Kc/s.}$$

Three such stages are connected in tandem to give 36 db/octave cut-off characteristics at the high cut-off end.

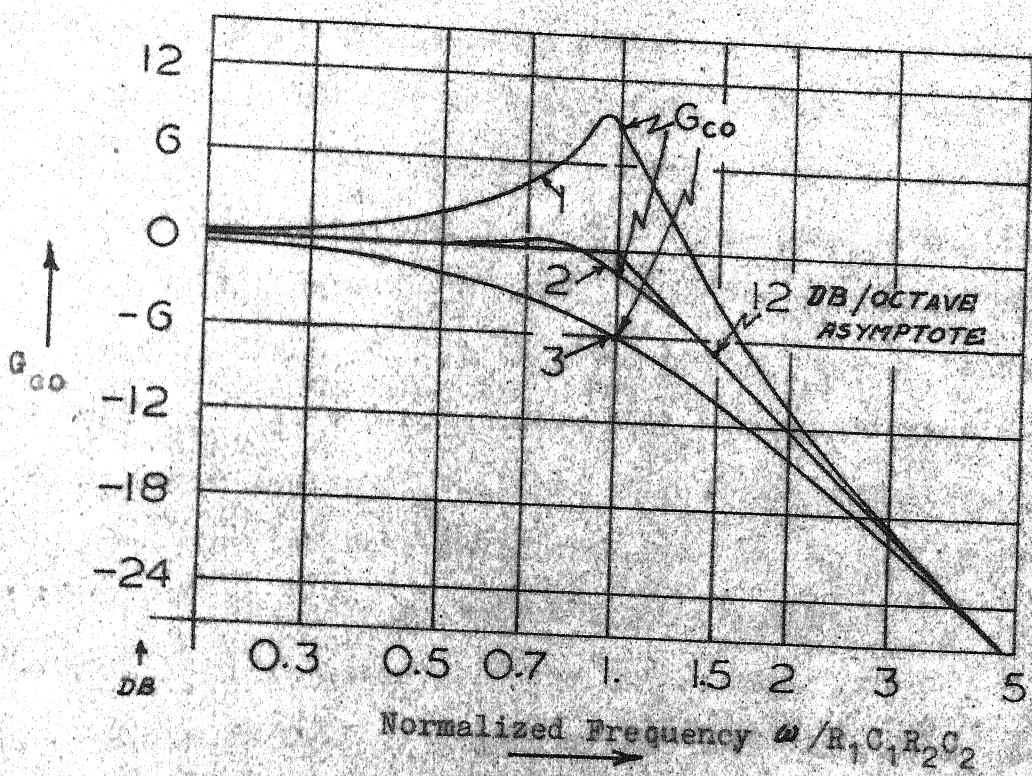


FIG. 13

PLOT OF  $G_{co}$  VERSUS FREQUENCY

The minimum frequency of undesirable harmonic contents to be rejected by the filter is 6.5 Mc/s. The filter response at 6.5 Mc/s, with 36 db/octave characteristic, is down by 720 db. The output of the filter is fed to the discriminator.

#### 4.44 Discriminator:

Fig. (14) shows the circuit diagram of the discriminator unit. The stage  $T_1$  amplifies the output of the active filter to sufficient amplitude to drive the switching transistor  $T_2$  between its cut-off and saturation states. Normally transistor  $T_2$  rests in the saturated state until driven by the signal to cut-off. The collector potential of  $T_2$  rises from saturation value of -0.4 volt to about -10 volts, which is clamped by diode  $D_2$ . The clamping diode  $D_2$  is fed from the emitter of transistor  $T_3$  which acts as a d.c. emitter follower and so provides a very low impedance source of voltage. The clamping voltage can be adjusted by varying the base resistors  $R_7$  and  $R_8$  of the transistor  $T_3$ .

The clipped output from transistor  $T_2$  is differentiated by a CR differentiator. The time constant of the differentiator is given by  $C_5R_{10}$ , and is of the order of 0.3 micro-seconds. This is very small in comparison to the time between pulses, which is 5 micro-seconds and corresponds to maximum possible signal frequency of 200 Kc/s. Diode  $D_3$  clips the negative spikes obtained in the process of differentiation.

Positive spikes are now fed to an a.c. coupled emitter follower  $T_4$ . This avoids the loading of the one shot (monostable multivibrator) input circuit.<sup>13</sup> The spikes thus obtained from the emitter follower are fed to the base of the transistor  $T_5$  of the one shot stage. With no triggering signal the transistor  $T_5$

stays in cut-off and the transistor  $T_6$  remains in saturation. As  $T_5$  receives the triggering signal, it turns on and is driven into saturation, and as a result of this  $T_6$  is cut off. This is the start of the quasi-stable state. The duration of the quasi-stable state can be adjusted by varying time constant  $C_8R_{18}$  which is done by inserting various values of the capacitor  $C_8$ . The detail design of the one shot is discussed elsewhere in the literature . The output of the one shot is well shaped and timed, and is taken from collector of the transistor  $T_6$ . To avoid loading of the one shot an emitter follower stage  $T_7$  is incorporated and the output is taken from its emitter. This output drives the frequency counter (Hewlett-Packard), an averaging d.c. meter and a rate control circuit.

The complete circuit diagram of the film thickness measuring unit is shown in Fig. (15).

## CHAPTER FIVE

### OBSERVATIONS AND RESULTS

Pressure as measured right on the top of the manifold port of the base plate without bell-jar is of the order of  $4 \times 10^{-8}$  torr. This is attained on pumping for two hours. When measured inside the evaporation chamber (15" dia., 25" high) the order of pressure is about  $10^{-7}$  torr after an hour of pumping, and without baking the system. However, the pressure continues to fall gradually with time. All pressure measurements have been taken with Veeco ionization gauge with a glass tubing of limited conductance which connects it to the base plate of the evaporation chamber.

Evaporation is started when the pressure has reached  $10^{-7}$  torr range. Ultra-high pure copper kept inside a molybdenum boat is electrically heated. The current in the boat is adjusted by controlling the primary voltage of the step down transformer with the help of a variable auto-transformer. The temperature is measured by means of an optical pyrometer (Leeds-Northrup type) to an accuracy of 20 °C.

The frequency of the signal from the output of the crystal monitor unit is observed on Hewlett-Packard electronic counter. Before starting the deposition of the thin film the initial frequency of the signal is recorded. The push-pull shutter is now operated for a certain measure of time during which copper vapours deposit on the substrate (microscopic glass plate) as well as on the quartz crystal. The thickness of the film

and the rate of deposition are calculated from the difference in the two frequency readings, (initial and at the end of the deposition) and the time of deposition.

For a typical run following are the observations and results:

Pressure in the evaporation chamber	= $10^{-7}$ torr
Setting of the auto-transformer at the time of evaporation	= 75 volts
Current in the primary of step down transformer	= 1 amp.
Temperature of the copper in the boat	= 1250 °C
Secondary tapping of the step down transformer	= 5 volts
Frequency of the counter at the time when deposition starts	= 22.54 kc/s
Frequency of the counter at the end of deposition	= 23.17 kc/s
Time of deposition	= 2 min.
Frequency shift	= 630 c/s
Area of the monitor crystal exposed to the evaporant	= 0.38 sq cm

Results:

Total mass deposited	= $8.6 \times 10^{-6}$ gm
Thickness of the film deposited	= 253 Å
Rate of deposition of the film	= 2.1 Å/sec.

CONCLUSIONS

A shift of 10 c/s has been observed in the frequency of resonance of the monitor crystal for a mass build up of  $3.6 \times 10^{-7}$  gm/sq cm of copper on the face of the crystal exposed to the evaporant. In terms of the thickness of the film this shift corresponds to a film thickness of  $4 \text{ \AA}$ . The differential weighing sensitivity of the quartz crystal monitor, defined as the ratio of the frequency shift to the mass deposited, is about 73 c/s/microgram. Thus, using a Hewlett Packard electronic counter it is possible to monitor mass changes of the order of  $10^{-8}$  gm with the help of the quartz crystal monitor unit. Film thickness of the order of 3 micron could be measured.



FUTURE WORK

Future work on the UHV evaporation unit would involve the following:-

- (i) providing additional facilities in the evaporation unit in order to make it more versatile;
- (ii) fabrication of microelectronic components and circuits;
- (iii) studying physical properties of the thin film deposited under various conditions.

The facilities which could be incorporated are: (i) an automatic feedback control system for the control of a.c. power source, supplying power to the heater (boat), to obtain constant evaporation rates during deposition of the thin film;<sup>14</sup> (ii) an arrangement for the deposition of alloy film of desired composition, for instance, film of permalloy (20% Fe, 80% Ni); (iii) baking arrangement for the evaporation chamber to attain still better vacuum.

Electronic components which could be fabricated are surface barrier diodes and insulated-gate field effect transistors, passive elements such as resistors, inductors and capacitors, and passive microcircuits. Thin film magnetic memories for computers,  $\text{Nb}_3\text{Sn}$  superconducting thin film yielding very high critical current densities,<sup>15</sup> and various other devices with improved performance could be realized after introducing above mentioned facilities for the deposition of alloy film.

Studying the effects of the growth conditions, in which thin film is deposited, on its structure and physical properties make up the third category. The structural details of the thin film are studied with the help of electron micrographs.

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